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(19) (11) Publication number: 1

PATENT ABSTRACTS OF JAPAN

(21) Application number: **10106014**

(51) Intl. Cl.: **C08G 77/14** H01B 1/12

(22) Application date: **16.04.98**

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(43) Date of application publication: **02.11.99**

(84) Designated contracting states:

(71) Applicant: **KANEGAFUCHI CHEM LTD**

(72) Inventor: **OKADA KENJI**

(74) Representative:

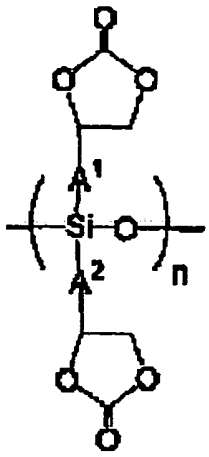
(54) POLYSILOXANE HAVING CYCLIC CARBONATE GROUP ON SIDE CHAIN AND ITS PRODUCTION

(57) Abstract:

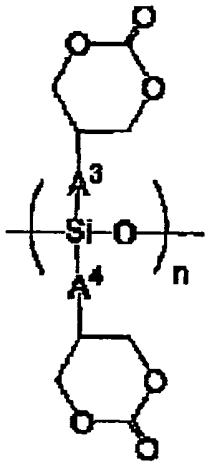
PROBLEM TO BE SOLVED: To efficiently obtain a polysiloxane having a substituent group containing a cyclic carbonate on the silicon and useful for polymer solid electrolyte, or the like, usable for battery, or the like.

SOLUTION: This polysiloxane has two substituent groups each containing a cyclic carbonate on their each silicon and preferably contains siloxane unit. The main chain is preferably represented by formula I [A1 and A2 are each an alkyl, an aryl or the like; (n) is an integer of ≥1], formula II[A3 and A4 are each an alkyl, an aryl or the like] or the like. The polysiloxane is obtained by reacting 1 mol dichlorosilane derivative of formula III (X1 and X2

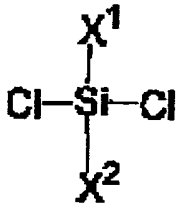
are each a cyclic carbonate) with preferably 0.5-4.0 mol zinc oxide, preferably in the presence of an organic solvent, preferably at 50-150° C, adding water and a protonic acid to the resultant reaction mixture and stirring these components to produce a diorganopolysiloxane having silanol groups at both ends of molecular chain and subjecting the diorganopolysiloxane to polycondensation in the presence of an acidic condensation catalyst, preferably at 10-150°C.



I



II



III

(書誌+要約+請求の範囲)

(19)【発行国】日本国特許庁(JP)
(12)【公報種別】公開特許公報(A)
(11)【公開番号】特開平11−302383
(43)【公開日】平成11年(1999)11月2日
(54)【発明の名称】環状カーボネート基を側鎖に有するポリシロキサン及びその製造方法
(51)【国際特許分類第6版】

C08G 77/14
H01B 1/12
// H01M 6/18
10/40

【FI】

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H01B 1/12 Z
H01M 6/18 E
10/40 B

【審査請求】未請求
【請求項の数】9
【出願形態】OL
【全頁数】9
(21)【出願番号】特願平10−106014
(22)【出願日】平成10年(1998)4月16日
(71)【出願人】
【識別番号】000000941
【氏名又は名称】鐘淵化学工業株式会社
【住所又は居所】大阪府大阪市北区中之島3丁目2番4号
(72)【発明者】
【氏名】岡田 賢治
【住所又は居所】兵庫県神戸市兵庫区吉田町1—2—80鐘淵化学工業株式会社神戸研究所内

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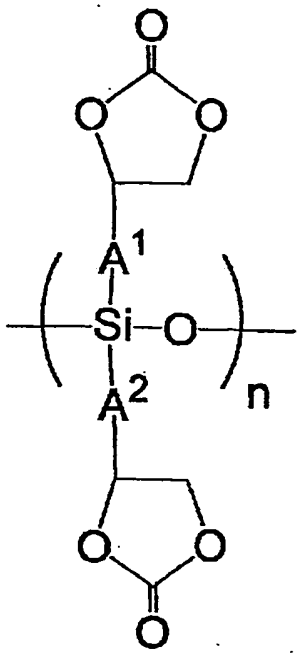
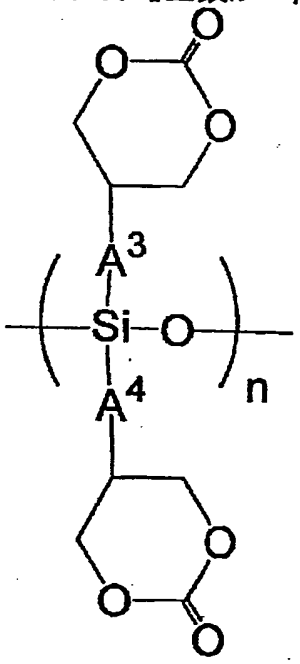
(57)【要約】
【課題】低温におけるイオン伝導度、強度などに優れた、各けい素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンを提供する。
【解決手段】ジクロロシランに対し、環状カーボネート構造を有する末端ビニル化合物を白金化合物触媒によりヒドロシリル化させ、得られたジクロロシラン誘導体を縮合重合させることにより各けい素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンを得る。

【特許請求の範囲】
【請求項1】各ケイ素上に環状カーボネートを含有する置換基を2つ有するポリシロキサン。
【請求項2】各ケイ素上に環状カーボネートを含有する置換基を2つ有するシロキサン単位を含むポリシロキサン。
【請求項3】主鎖が一般式【化1】



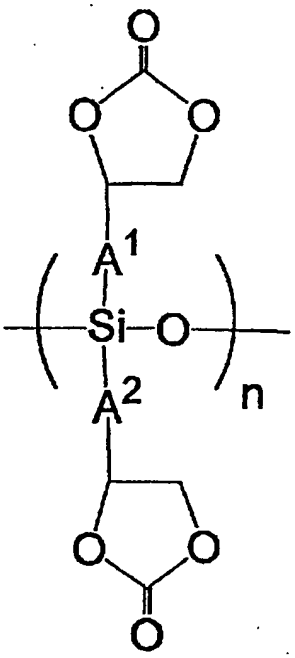
で示される、各ケイ素上に環状カーボネートを含有する置換基を2つ有する請求項1に記載のポリシロキサン。(式中A^1、A^2は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、nは1以上の整数を表す)

【請求項4】主鎖が一般式【化2】



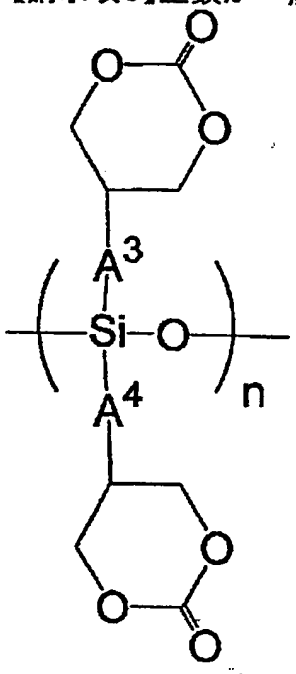
で示される、各ケイ素上に環状カーボネートを含有する置換基を2つ有する請求項1に記載のポリシロキサン。(式中A^3、A^4は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、nは1以上の整数を表す)

【請求項5】主鎖が一般式【化3】



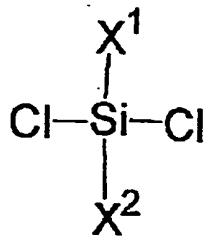
で示される、各ケイ素上に環状カーボネートを含有する置換基を2つ有するシロキサン単位を含有する請求項2に記載のポリシロキサン。(式中A^1、A^2は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、nは1以上の整数を表す)

【請求項6】主鎖が一般式【化4】

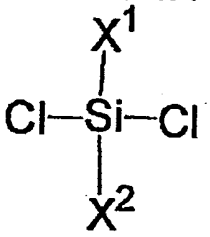


で示される、各ケイ素上に環状カーボネートを含有する置換基を2つ有するシロキサン単位を含有する請求項2に記載のポリシロキサン。(式中A^3、A^4は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、nは1以上の整数を表す)

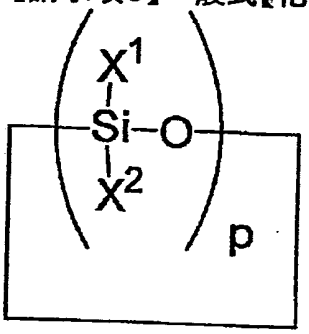
【請求項7】一般式【化5】



(式中X^1、X^2は環状のカーボネート基を有する置換基であり、X^1、X^2はそれぞれ同種または異種であってもよい)で示されるジクロロシラン誘導体と、酸化亜鉛を有機溶媒の存在下で反応させ、次いで、得られた反応混合物に水およびプロトン酸を加えて撹拌することにより、分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に、該ジオルガノポリシロキサンを酸性縮合触媒の存在下に縮合重合させることを特徴とする、請求項1に記載の各けい素上に環状カーボネートを含有する置換基を2個有するポリシロキサンの製造方法【請求項8】一般式【化6】



(式中X^1、X^2は環状のカーボネート基を有する置換基であり、X^1、X^2はそれぞれ同種または異種であってもよい)で示されるジクロロシラン誘導体を、酸を触媒として溶媒の存在下に混合することにより分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に、該ジオルガノポリシロキサンを酸性縮合触媒の存在下に縮合重合させることを特徴とする、請求項1に記載の各けい素上に置換基として環状カーボネート基を2個有するポリシロキサンの製造方法。
【請求項9】一般式【化7】



(式中X^1、X^2は環状のカーボネート基を有する置換基であり、X^1、X^2はそれぞれ同種または異種であってもよく、pは3または4)で示されるシクロシロキサン誘導体を、プロトン酸触媒の存在下に重合させることを特徴とする、請求項1に記載の各けい素上に置換基として環状カーボネート基を2個有するポリシロキサンの製造方法。

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(11)Publication number : 11-302383
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H01B 1/12
// H01M 6/18
H01M 10/40

(21)Application number : 10-106014
(22)Date of filing : 16.04.1998

(71)Applicant : KANEGAFUCHI CHEM IND CO LTD
(72)Inventor : OKADA KENJI

(54) POLYSILOXANE HAVING CYCLIC CARBONATE GROUP ON SIDE CHAIN AND ITS PRODUCTION

(57)Abstract:
PROBLEM TO BE SOLVED: To efficiently obtain a polysiloxane having a substituent group containing a cyclic carbonate on the silicon and useful for polymer solid electrolyte, or the like, usable for battery, or the like.
SOLUTION: This polysiloxane has two substituent groups each containing a cyclic carbonate on their each silicon and preferably contains siloxane unit. The main chain is preferably represented by formula I [A1 and A2 are each an alkyl, an aryl or the like; (n) is an integer of ≥1], formula II[A3 and A4 are each an alkyl, an aryl or the like] or the like. The polysiloxane is obtained by reacting 1 mol dichlorosilane derivative of formula III (X1 and X2 are each a cyclic carbonate) with preferably 0.5-4.0 mol zinc oxide, preferably in the presence of an organic solvent, preferably at 50-150°C, adding water and a protonic acid to the resultant reaction mixture and stirring these components to produce a diorganopolysiloxane having silanol groups at both ends of molecular chain and subjecting the diorganopolysiloxane to polycondensation in the presence of an acidic condensation catalyst, preferably at 10-150°C.

I

II

III

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted

registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

(19)日本国特許庁（JP）　(12)公開特許公報（A）　(11)特許出願公開番号

特開平11－302383

(43)公開日　平成11年(1999)11月2日

(51)Int.Cl.⁶	識別記号	FI		
C08G 77/14		C08G	77/14	
H01B 1/12		H01B	1/12	Z
// H01M 6/18		H01M	6/18	E
10/40			10/40	B

審査請求　未請求　請求項の数9　OL　（全 9 頁）

(21)出願番号　特願平10－106014

(22)出願日　平成10年(1998)4月16日

(71)出願人　000000941
鐘淵化学工業株式会社
大阪府大阪市北区中之島3丁目2番4号

(72)発明者　岡田　貴治
兵庫県神戸市兵庫区吉田町1－2－80鐘淵化学工業株式会社神戸研究所内

(54)【発明の名称】　環状カーボネート基を側鎖に有するポリシロキサン及びその製造方法

(57)【要約】
【課題】　低温におけるイオン伝導度、強度などに優れた、各けい素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンを提供する。
【解決手段】　ジクロロシランに対し、環状カーボネート構造を有する末端ビニル化合物を白金化合物触媒によりヒドロシリル化させ、得られたジクロロシラン誘導体を縮合重合させることにより各けい素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンを得る。

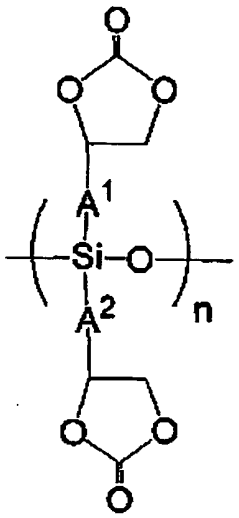
【特許請求の範囲】

【請求項１】各ケイ素上に環状カーボネートを含有する置換基を２つ有するポリシロキサン。

【請求項２】各ケイ素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含むポリシロキサン。

【請求項３】主鎖が一般式

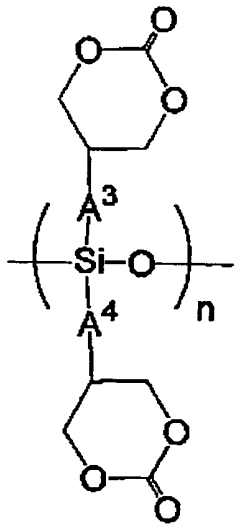
【化１】



で示される、各ケイ素上に環状カーボネートを含有する置換基を２つ有する請求項１に記載のポリシロキサン。（式中A'、A'は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、ｎは１以上の整数を表す）

【請求項４】主鎖が一般式

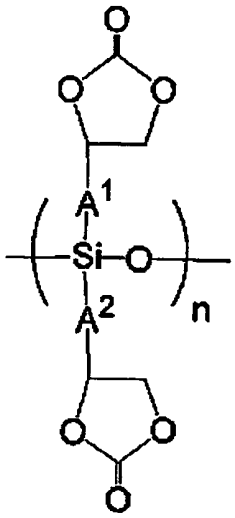
【化２】



で示される、各ケイ素上に環状カーボネートを含有する置換基を２つ有するポリシロキサン。（式中A'、A'は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、ｎは１以上の整数を表す）

【請求項５】主鎖が一般式

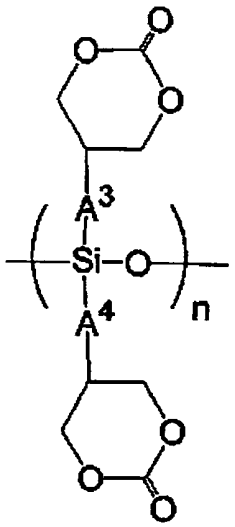
【化３】



で示される、各ケイ素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含有する請求項２に記載のポリシロキサン。（式中A¹、A²は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、ｎは１以上の整数を表す）

【請求項６】主鎖が一般式

【化４】

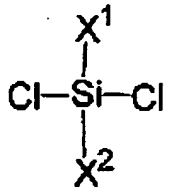


で示される、各ケイ素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含有する請求項２に記載のポリシロキサン。（式中A'、A'は、置換また

は非置換のアルキル基、アリール基、またはオキシアルキレン基を、nは1以上の整数を表す）

【請求項7】一般式

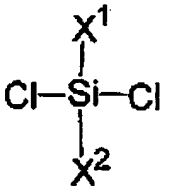
【化5】



（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシラン誘導体と、酸化亜鉛を有機溶媒の存在下で反応させ、次いで、得られた反応混合物に水およびプロトン酸を加えて撹拌することにより、分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に、該ジオルガノポリシロキサンを酸性縮合触媒の存在下に縮合重合させることを特徴とする、請求項1に記載の各けい素上に環状カーボネートを含有する置換基を2個有するポリシロキサンの製造方法

【請求項8】一般式

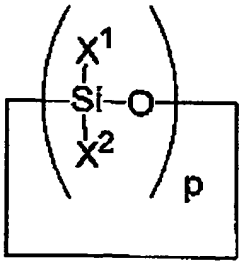
【化6】



（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシラン誘導体を、酸を触媒として溶媒の存在下に混合することにより分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に、該ジオルガノポリシロキサンを酸性縮合触媒の存在下に縮合重合させることを特徴とする、請求項1に記載の各けい素上に置換基として環状カーボネート基を2個有するポリシロキサンの製造方法。

【請求項9】一般式

【化7】



（式中X¹、X²は環状のカーボネート基を有する置換基

であり、X¹、X²はそれぞれ同種または異種であってもよく、pは3または4）で示されるシクロシロキサン誘導体を、プロトン酸触媒の存在下に重合させることを特徴とする、請求項1に記載の各けい素上に置換基として環状カーボネート基を2個有するポリシロキサンの製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は電池などに使用可能な高分子固体電解質などになりうる各けい素上に環状カーボネートを含有する置換基を2つ有するポリシロキサンおよびその製造方法に関する。

【0002】

【従来の技術】近年、樹脂工業が発展するとともに、特殊な性質を有する樹脂が必要になり、それに伴って従来にない高分子の開発が望まれるようになってきた。エポキシ化合物の開環重合により得られるポリエーテルは、特殊ゴム、界面活性剤など、その用途は広い。またポリエチレンオキシドを側鎖に持つ高分子は、リチウム塩などのアルカリ金属塩を可溶化することができ、高分子固体電解質への研究が盛んである。しかしながら、ポリエチレンオキシドはその高い結晶性のために、金属塩の移動度を十分に向上させることができない。

【0003】

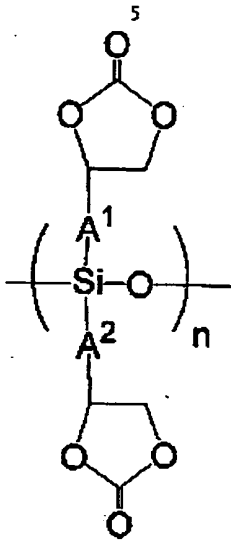
【発明が解決しようとする課題】本発明は、各ケイ素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンおよびその製造方法を提供することにある。

【0004】

【課題を解決するための手段】本発明は、各ケイ素上に環状カーボネートを含有する置換基を2つ有する新規なポリシロキサンおよびその製造方法を提供する。好ましくは、主鎖が一般式

【0005】

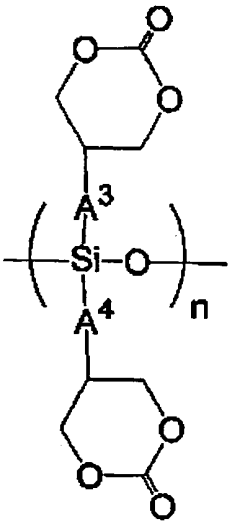
【化8】



【０００６】で示される．各ケイ素上に環状エーテルを含有する置換基を２つ有する上記記載のポリシロキサン（式中A'．A"は、置換または非置換のアルキル基、アリール基、またはオキシアルキレン基を、ｎは１以上の整数を表す）。また好ましくは、主鎖が一般式

【０００７】

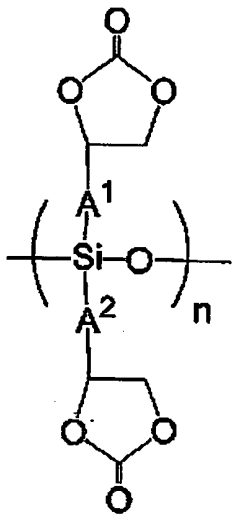
【化９】



【０００８】で示される．各ケイ素上に環状カーボネートを含有する置換基を２つ有する上記記載のポリシロキサン（式中A'．A"は、置換または非置換のアルキル基．アリール基．またはオキシアルキレン基を．ｎは１以上の整数を表す）。また各けい素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含むポリシロキサンおよびその製造方法に関する。かかる各ケイ素上に２つの環状カーボネートを含有する置換基を有するポリシロキサンは．少量の他のシロキサン単位、例えば$R_2SiO_{2/2}$単位（式中、Rは置換あるいは非置換のアルキル基である）．$SiO_{4/2}$単位を含有しても良い。好ましいものとして．主鎖が一般式

【０００９】

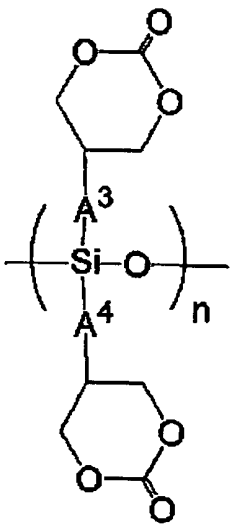
【化１０】



【００１０】で示される．各ケイ素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含有する上記記載のポリシロキサン（式中A'．A"は．置換または非置換のアルキル基．アリール基、またはオキシアルキレン基を．ｎは１以上の整数を表す）を挙げることができる。また好ましいものとして．主鎖が一般式

【００１１】

【化１１】

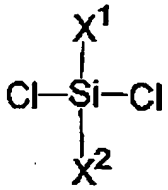


【００１２】で示される．各ケイ素上に環状カーボネートを含有する置換基を２つ有するシロキサン単位を含有する上記記載のポリシロキサン（式中A'．A"は．置換

または非置換のアルキル基．アリール基、またはオキシアルキレン基を．ｎは１以上の整数を表す）を挙げることができる。上記ポリシロキサンの製造法は例えば次に示す方法である。一般式

【００１３】

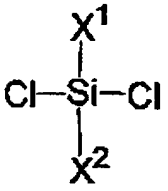
【化１２】



【００１４】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシラン誘導体を．プロトン酸を触媒として水の存在下に攪拌することにより、分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に該ジオルガノポリシロキサンを酸性縮合触媒存在下に縮合重合させることを特徴とする、前記各ケイ素上に環状カーボネートを含有する置換基を２つ有する新規なポリシロキサンおよびその製造方法に関する。また本発明は、一般式

【００１５】

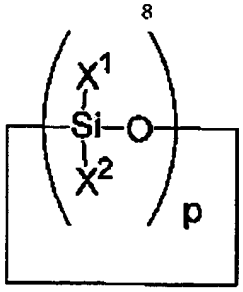
【化１３】



【００１６】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシラン誘導体を．酸化亜鉛を有機溶媒の存在下で反応させ、次いで得られた反応混合物に、例えば塩酸などで代表されるプロトン酸を触媒として水の存在下に攪拌することにより、分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、しかる後に該ジオルガノポリシロキサンを酸性縮合触媒存在下に縮合重合させることを特徴とする、前記各ケイ素上に環状カーボネート基を含有する置換基を２つ有する新規なポリシロキサンおよびその製造方法に関する。さらに本発明は、一般式

【００１７】

【化１４】



【００１８】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよく、ｐは３または４）で示されるシクロシロキサン誘導体を、プロトン酸触媒の存在下に重合させることを特徴とする前記各ケイ素上に環状カーボネートを含有する置換基を２つ有する新規なポリシロキサンの製造方法に関する。

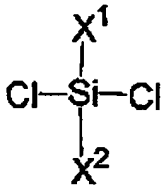
【００１９】

【発明の実施の形態】本発明の製造方法を実施するにあたっては、該ジクロロシランに対して酸化亜鉛あるいはプロトン酸を適当な反応容器中で接触させる。本発明においては反応容器の種類は重要でない。しかしながら副反応を防ぐため．非反応性材料で形成された反応容器中でおこなうのが好ましい。本発明方法は、バッチ法、セミバッチ法または連続式で実施しうる。この反応容器は．例えば連続的攪拌タンク反応容器でありうる。この方法はバッチ式あるいは連続式でおこなうのが好ましい。

一般式

【００２０】

【化１５】

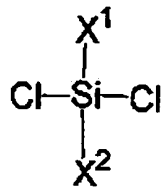


【００２１】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシランに対して酸化亜鉛を有機溶媒の存在下で接触あるいはプロトン酸を接触させるが．その接触方法に特に制約はない。本発明の各ケイ素上に環状カーボネートを含有する置換基を２つ有するポリシロキサンは、一般式

【００２２】

【化１６】

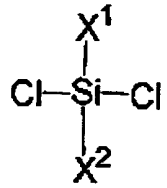
9



【0023】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で表されるジクロロシランと酸化亜鉛を有機溶媒の存在下で反応させ、次いで得られた反応混合物に水およびプロトン酸を加えて撹拌することにより、分子鎖両末端にシラノール基を有するジオルガノポリシロキサンを生成せしめ、次いで該ジオルガノポリシロキサンを酸性縮合触媒の存在下に縮合重合させるのであるが、始発原料である一般式

【0024】

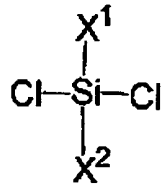
【化17】



【0025】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で示されるジクロロシランは例えば次のような方法で製造される。ジクロロシラン（H_2SiCl_2）および環状カーボネート基を含有する末端ビニル化合物を、ヒドロシリル化反応用触媒である白金化合物触媒あるいはロジウム化合物触媒と接触させ付加反応をすることにより得られる。この種のジクロロシランを合成する別の方法としては、例えば、テトラクロロシラン（$SiCl_4$）に対して相当するグルニヤール反応剤を反応させて得る方法がある。本発明の各ケイ素上に環状カーボネートを含有する置換基を2つ有するポリシロキサンは前記したような方法で製造された、一般式

【0026】

【化18】



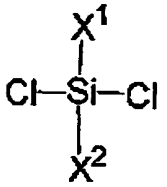
【0027】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で表されるジクロロシランと酸化亜鉛を有機溶媒の存在下で反応させ、次いで、得られた反応混合物に水およびプロトン酸を加えて撹拌することにより、分子末端にシラノール基を有するジオルガノポリ

10

シロキサンを生成せしめるのであるが、ここで酸化亜鉛とジクロロシランは有機溶媒中で反応し、対応するポリシロキサンと副生物として塩化亜鉛を生じる。反応温度は特に限定はされないが、好ましくは－30℃～200℃の範囲で実施され、さらに好ましくは50℃～150℃の過熱下で実施される。ここで酸化亜鉛の使用量は、通常、上記ジクロロシラン1モル当たり、0.4～5.0モルとなるような量であり、当量である0.5から4.0モルが好ましい。本発明に使用される有機溶媒は、アセトニトリル、ケトンおよび酢酸アルキルが挙げられる。酢酸アルキルとしては、酢酸メチル、酢酸エチル、酢酸イソプロピル、酢酸n－プロピル、酢酸イソブチルなどが例示され、ケトンとしてはメチルエチルケトン、メチルイソブチルケトンが例示されるが、好ましくはアセトニトリル、酢酸メチル、酢酸エチル、酢酸イソブチル、メチルイソブチルケトンが用いられる。またこれらの有機溶媒に、他の有機溶媒を併用することも可能である。引き続いて、水とプロトン酸を加えることにより、分子鎖末端にシラノール基を有するポリシロキサンが生成され、このものは有機溶媒層に溶解し、副生物としての塩化亜鉛は水層に溶解し、また、過剰の酸化亜鉛も水層に溶解して2層に分離される。有機層は純水で洗浄を繰り返した後、回収、脱水される。使用した溶媒は減圧除去等の方法で取り除く。さらに酸性縮合触媒を用いて分子量を増大させることが出来る。酸性縮合触媒としては、濃硫酸、トリフルオロメタンスルホン酸、ドデシルベンゼンスルホン酸などが例示される。また、2－エチルヘキサン酸をテトラメチルグアニジンと当モルで混合したものも有効である。室温で縮合反応を起こしシロキサン鎖の切断を起こしにくいので、好ましくはトリフルオロメタンスルホン酸を使用する。縮合反応の温度範囲は特に限定されるものではないが、好ましくは10℃から150℃の温度範囲で実施される。また本発明の各ケイ素上に環状カーボネートを含有する置換基を2つ有するポリシロキサンは前記したような方法で製造された、一般式

【0028】

【化19】



【0029】（式中X¹、X²は環状のカーボネート基を有する置換基であり、X¹、X²はそれぞれ同種または異種であってもよい）で表されるジクロロシランと酸を有機溶媒の存在下で反応させ、次いで、得られた反応混合物に水およびプロトン酸を加えて撹拌することにより、分子末端にシラノール基を有するジオルガノポリシロキ

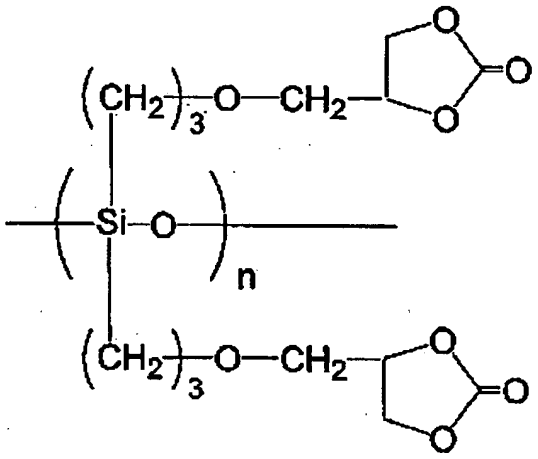
サンを生成せしめるのであるが、ここで使用する酸は特に限定されるものではないが、好ましくは塩酸などが挙げられる。ここで酸の使用量は、通常、上記ジクロロシラン1モル当たり、0.4～5.0モルとなるような量であり、当量である0.5から4.0モルが好ましい。本発明に使用される有機溶媒は、ジエチルエーテル、アセトニトリル、ケトンおよび酢酸アルキルが挙げられる。酢酸アルキルとしては、酢酸メチル、酢酸エチル、酢酸イソプロピル、酢酸n-プロピル、酢酸イソブチルなどが例示され、ケトンとしてはメチルエチルケトン、メチルイソブチルケトンなどが例示されるが、好ましくはメチルエチルケトンやメチルイソブチルケトンが用いられる。またこれらの有機溶媒に、他の有機溶媒を併用することも可能である。こうして得られた分子鎖末端にシラノール基を有するポリシロキサンは、酸性縮合触媒＊

＊存在下にさらに重合することが出来る。酸性縮合触媒としては、濃硫酸、トリフルオロメタンスルホン酸、ドデシルベンゼンスルホン酸などが例示される。また、2-エチルヘキサン酸をテトラメチルグアニジンと当モルで混合したものも有効である。室温で縮合反応を起こしシロキサン鎖の切断を起こしにくいので、好ましくはトリフルオロメタンスルホン酸を使用する。縮合反応の温度範囲は特に限定されるものではないが、好ましくは室温から150℃の温度範囲で実施される。このような方法により得られる該ポリシロキサンの化合物例を示すが、本発明は以下の化合物に限定されるものではない。なお化合物1～6において、nは1以上の整数を表す。

（化合物例1）

【0030】

【化20】

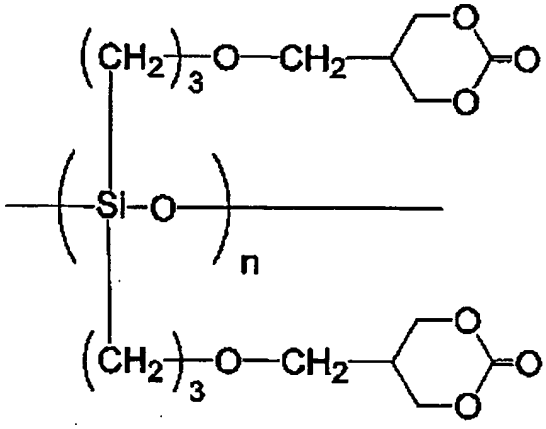


【0031】（化合物例2）

【0032】

※【化21】

※



【0033】（化合物例3）

【0034】

【化22】

い。

（実施例１）ガラス内筒を備えたステンレス製オートクレーブに、環状（３－ブテニル）エチレンエステルカルボン酸１４．２ｇおよび白金ビニルシロキサンキシレン溶液１．０ｍｇ（１．０×１０$^{-4}$ｍｍｏｌ）を秤取し、空気中でそのオートクレーブに、ジクロロシラン２．１５ｇを加え、６０℃に加熱して攪拌した。６時間後反応を終了し反応混合物をクーゲルロールを使用して蒸留した。その結果ケイ素上に環状カーボネート基を２つ有するジクロロシラン誘導体が．５．６５ｇ（１４．７ｍｍｏｌ．収率８８％）得られた。またこの物質のＧＣ－ＭＳによる分子量測定では親ピークが３８５に現れた。次に得られたジクロロシラン誘導体３．８４ｇを．酸化亜鉛１．２２ｇ．酢酸エチル５．０ｇの入った反応容器に．６０℃に加熱攪拌下で１時間かけて滴下した。その

後さらに３時間加熱攪拌を行なった。冷却後純水２０ｇを加え、さらに１０％塩酸水をフラスコの固形分がなくなるまで添加した。有機層を取り出し純水で十分洗浄したのち、有機溶媒を減圧除去することによりポリシロキサン３．１５ｇを得た。このポリシロキサン０．８ｇと過塩素酸リチウム０．２ｇとをアセトンに溶解させ均一溶液とし、それを基板上に流延した後．アルゴンガス雰囲気下で加熱して溶媒を除去し薄膜を得た。この薄膜のイオン伝導度は極めて高い値を示した。

【００４２】

【発明の効果】本発明の各ケイ素上に環状カーボネートを含有する置換基を２つ有するポリシロキサンは新規な化合物であり．またその製造方法は簡便で効率が良いという特徴を有する。

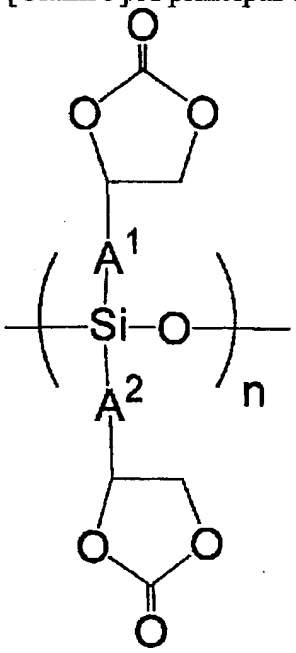
CLAIMS

[Claim(s)]

[Claim 1] The polysiloxane which has two substituents which contain annular carbonate on each silicon.

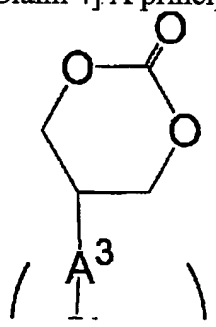
[Claim 2] A polysiloxane including the siloxane unit which has two substituents which contain annular carbonate on each silicon.

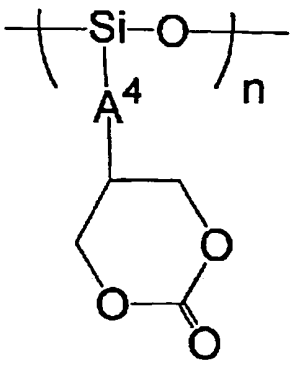
[Claim 3] A principal chain is a general formula. [Formula 1]



The polysiloxane according to claim 1 which comes out and has two substituents which are shown, and which contain annular carbonate on each silicon. (As for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine)

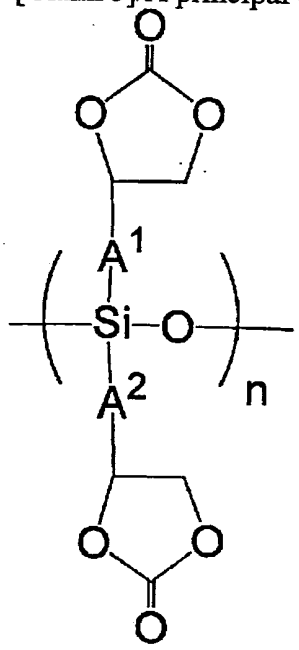
[Claim 4] A principal chain is a general formula. [Formula 2]





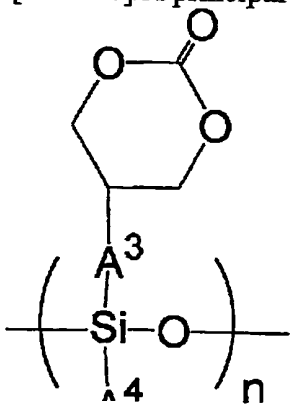
The polysiloxane according to claim 1 which comes out and has two substituents which are shown, and which contain annular carbonate on each silicon. (As for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine)

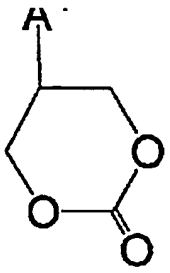
[Claim 5] A principal chain is a general formula. [Formula 3]



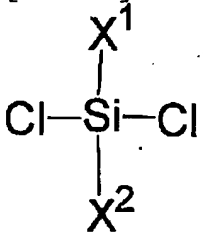
The polysiloxane according to claim 2 which comes out and contains the siloxane unit which is shown, and which has two substituents which contain annular carbonate on each silicon. (As for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine)

[Claim 6] A principal chain is a general formula. [Formula 4]

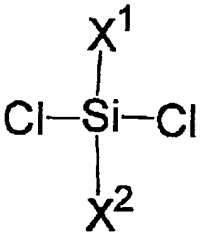




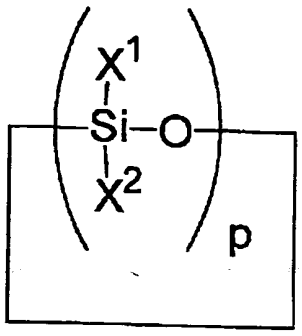
The polysiloxane according to claim 2 which comes out and contains the siloxane unit which is shown, and which has two substituents which contain annular carbonate on each silicon. (As for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine)
[Claim 7] General formula. [Formula 5]



JIO characterized by providing the following. It is the dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species). The manufacture method of a polysiloxane which chain both ends are made to generate a silanol-group luganot polysiloxane, and is characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of existence of an acid condensation catalyst by making a zinc oxide react under existence of an organic solvent, and subsequently adding and stirring water and a proton acid into the obtained reaction mixture of having two substituents which contain annular carbonate on each silicon according to claim 1. [Claim 8] General formula. [Formula 6]



The dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) Chain both ends are made to generate the diorganopolysiloxane which has a silanol group by mixing under existence of a solvent by making an acid into a catalyst. The manufacture method of a polysiloxane characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of existence of an acid condensation catalyst of having two annular carbonate machines as a substituent on each silicon according to claim 1.
[Claim 9] General formula. [Formula 7]



(-- the substituent which has a carbonate machine annular the inside X1 and X2 of a formula -- it is -- that X1 and X2 are of the same kind respectively or the manufacture method of a polysiloxane which may be different species and is characterized by p carrying out the polymerization of the cyclosiloxane derivative shown by 3 or 4) to the bottom of existence of a proton-acid catalyst of having two annular carbonate machines as a substituent on each silicon according to claim 1.

[Translation done.]

* NOTICES *

1. This document has been translated by computer.So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the polysiloxane which has two substituents which contain annular carbonate on a cell etc. on each silicon which may become an usable solid polymer electrolyte etc., and its manufacture method.

[0002]

[Description of the Prior Art] While resin industry develops in recent years, the resin which has a special property is needed and development of the macromolecule which is not in the former in connection with it has come to be desired. The uses of the polyether obtained by the ring opening polymerization of an epoxy compound, such as special rubber and a surfactant, are large. Moreover, the macromolecule which has a polyethylene oxide in a side chain can solubilize alkali-metal salts, such as lithium salt, and the research to a polyelectrolyte is prosperous in it. However, a polyethylene oxide cannot fully raise mobility of a metal salt for [the] high crystallinity.

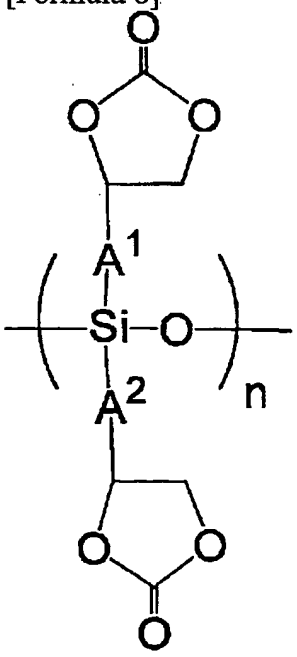
[0003]

[Problem(s) to be Solved by the Invention] this invention is to offer the new polysiloxane which has two substituents which contain annular carbonate on each silicon, and its manufacture method.

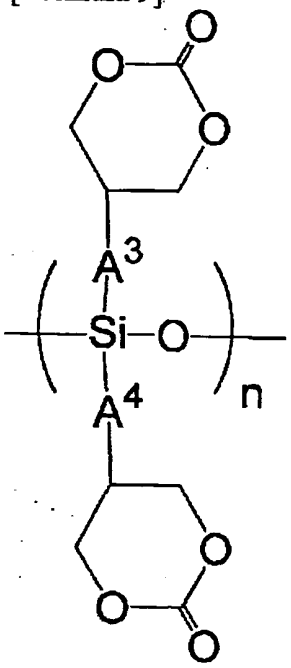
[0004]

[Means for Solving the Problem] this invention offers the new polysiloxane which has two substituents which contain annular carbonate on each silicon, and its manufacture method. Preferably, a principal chain is a general formula. [0005]

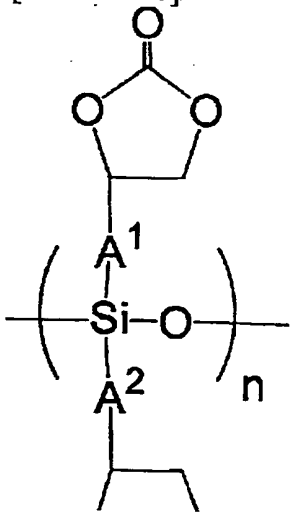
[Formula 8]

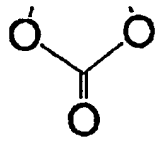


[0006] The polysiloxane of the above-mentioned publication which comes out and has two substituents which are shown, and which contain cyclic ether on each silicon (as for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine). Moreover, a principal chain is a general formula preferably.
[0007]
[Formula 9]



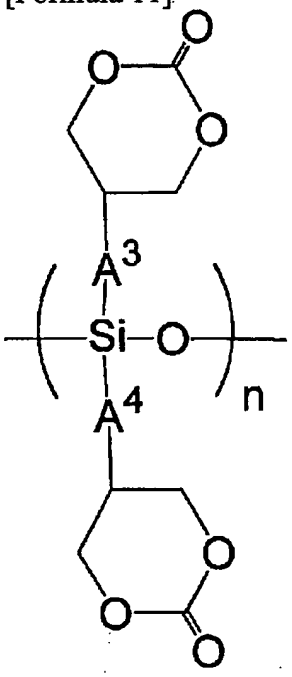
[0008] The polysiloxane of the above-mentioned publication which comes out and has two substituents which are shown, and which contain annular carbonate on each silicon (as for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine). Moreover, it is related with a polysiloxane including the siloxane unit which has two substituents which contain annular carbonate on each silicon, and its manufacture method. The polysiloxane which has the substituent which contains two annular carbonate on each of this silicon may contain other little siloxane units (the inside of a formula and R are substitution or an unsubstituted alkyl group), for example, R3SiO3/2 unit, and SiO4/2 unit. As a desirable thing, a principal chain is a general formula. [0009]
[Formula 10]





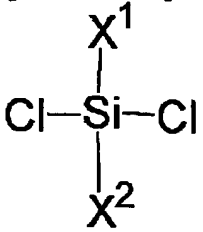
[0010] It can come out and the polysiloxane (as for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine) of the above-mentioned publication containing the siloxane unit which has two substituents which contain annular carbonate on each silicon shown can be mentioned. Moreover, as a desirable thing, a principal chain is a general formula. [0011]

[Formula 11]



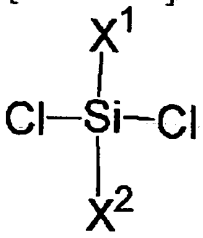
[0012] It can come out and the polysiloxane (as for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine) of the above-mentioned publication containing the siloxane unit which has two substituents which contain annular carbonate on each silicon shown can be mentioned. The manufacturing method of the above-mentioned polysiloxane is a method shown below. General formula. [0013]

[Formula 12]

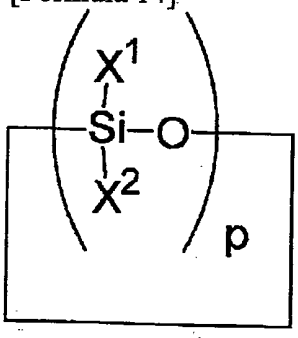


[0014] By stirring the dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) under existence of water by making a proton acid into a catalyst Chain both ends are made to generate the diorganopolysiloxane which has a silanol group. It is related with the new polysiloxane which is characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of acid condensation

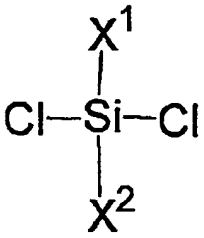
catalyst existence and which has two substituents which contain annular carbonate on each aforementioned silicon, and its manufacture method. Moreover, this invention is a general formula.
[0015]
[Formula 13]



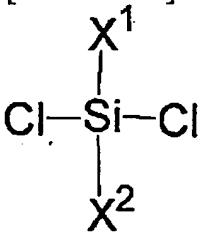
[0016] The dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) Into the reaction mixture which the zinc oxide was made to react under existence of an organic solvent, and was subsequently obtained, for example, by stirring under existence of water by making into a catalyst the proton acid represented with a hydrochloric acid etc. Chain both ends are made to generate the diorganopolysiloxane which has a silanol group. It is related with the new polysiloxane which is characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of acid condensation catalyst existence and which has two substituents which contain an annular carbonate machine on each aforementioned silicon, and its manufacture method. Furthermore, this invention is a general formula. [0017]
[Formula 14]



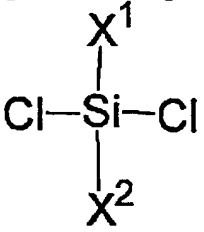
[0018] (-- the inside X1 and X2 of a formula is the substituent which has an annular carbonate machine, and is related with the manufacture method of that X1 and X2 are of the same kind respectively or the new polysiloxane which has two substituents which contain annular carbonate on each aforementioned silicon which may be different species and is characterized by p carrying out the polymerization of the cyclosiloxane derivative shown by 3 or 4) to the bottom of existence of a proton-acid catalyst
[0019]
[Embodiments of the Invention] If it hits enforcing the manufacture method of this invention, a zinc oxide or a proton acid is contacted in a suitable reaction container to this dichlorosilane. In this invention, the kind of reaction container is not important. However, in order to prevent side reaction, it is desirable to carry out in the reaction container formed with non-reactivity material. this invention method can be enforced with a batch method, a semi batch method, or continuous system. This reaction container is for example, a continuous stirring tank reaction container, and it deals in it. As for this method, it is desirable that a batch type or continuous system performs.
General formula. [0020]
[Formula 15]



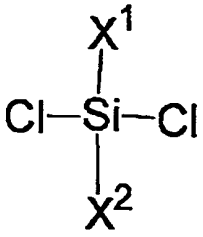
[0021] Although a zinc oxide contacts contact or a proton acid under existence of an organic solvent to the dichlorosilane shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species), there are especially no restrictions in the contact method. The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is a general formula.
[0022]
[Formula 16]



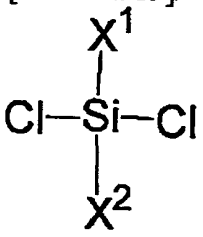
[0023] The dichlorosilane and the zinc oxide which are expressed with (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) are made to react under existence of an organic solvent. Subsequently, although chain both ends are made to generate the diorganopolysiloxane which has a silanol group and the condensation polymerization of this diorganopolysiloxane is carried out subsequently to the bottom of existence of an acid condensation catalyst by adding and stirring water and a proton acid into the obtained reaction mixture The general formula which is a first train raw material. [0024]
[Formula 17]



[0025] The dichlorosilane shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) is manufactured, for example by the following methods. It is obtained by contacting the end vinyl compound containing dichlorosilane (H_2SiCl_2) and an annular carbonate machine for the platinum compound catalyst or rhodium compound catalyst which is a catalyst for a hydrosilylation reaction, and carrying out an addition reaction. As an option which compounds this kind of dichlorosilane, there is the method of making the GURUNI yard reaction agent which corresponds to a tetrapod chlorosilicane ($SiCl_4$) react, and acquiring, for example. The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is the general formula manufactured by method which was described above. [0026]
[Formula 18]



[0027] The dichlorosilane and the zinc oxide which are expressed with (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) are made to react under existence of an organic solvent. subsequently Although a molecule end is made to generate the diorganopolysiloxane which has a silanol group by added and stirring water and a proton acid into the obtained reaction mixture A zinc oxide and dichlorosilane react in an organic solvent, and produce a zinc chloride as a corresponding polysiloxane and a corresponding by-product here. Although especially limitation is not carried out, reaction temperature is preferably carried out in -30 degrees C - 200 degrees C, and is carried out under 50 degrees C - 150 degrees C overheating still more preferably. The amount of the zinc oxide used usually has desirable 0.5 to 4.0 which is the amount which becomes 0.4-5.0 mols per one mol of above-mentioned dichlorosilane, and is the equivalent mols here. As for the organic solvent used for this invention, an acetonitrile, a ketone, and an acetic-acid alkyl are mentioned. Although methyl acetate, ethyl acetate, an isopropyl acetate, n propyl acetate, an isobutyl acetate, etc. are illustrated and a methyl ethyl ketone and a methyl isobutyl ketone are illustrated as a ketone as an acetic-acid alkyl, an acetonitrile, methyl acetate, ethyl acetate, an isobutyl acetate, and a methyl isobutyl ketone are used preferably. Moreover, it is also possible to use other organic solvents together to these organic solvents. Then, by adding water and a proton acid, the polysiloxane which has a silanol group at the chain end is generated, this thing is dissolved in an organic-solvent layer, and the zinc chloride as a by-product dissolves in a water layer, and a superfluous zinc oxide also dissolves in a water layer, and it separates into two-layer. After an organic layer repeats washing with pure water, it is collected and dehydrated. The used solvent is removed by methods, such as reduced pressure removal. Furthermore, molecular weight can be increased using an acid condensation catalyst. As an acid condensation catalyst, a concentrated sulfuric acid, a trifluoromethane sulfonic acid, dodecylbenzenesulfonic acid, etc. are illustrated. Moreover, what mixed 2-ethyl hexanoic acid with tetramethyl guanidine by this mol is effective. Since a condensation reaction is started at a room temperature and it is hard to start a siloxane chain scission, a trifluoromethane sulfonic acid is used preferably. Although especially the temperature requirement of a condensation reaction is not limited, it is preferably carried out by the 10 to 150 degrees C temperature requirement. Moreover, the polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is the general formula manufactured by method which was described above. [0028]
[Formula 19]



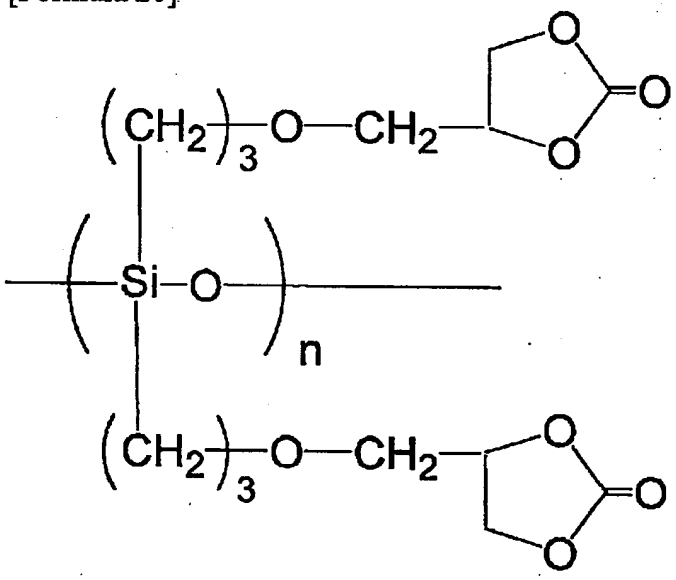
[0029] The dichlorosilane and the acid which are expressed with (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) are made to react under existence of an organic solvent. subsequently Although especially the acid used here although a molecule end is made to generate the

diorganopolysiloxane which has a silanol group by added and stirring water and a proton acid into the obtained reaction mixture is not limited, a hydrochloric acid etc. is mentioned preferably. The amount of the acid used usually has desirable 0.5 to 4.0 which is the amount which becomes 0.4-5.0 mols per one mol of above-mentioned dichlorosilane, and is the equivalent mols here. As for the organic solvent used for this invention, diethylether, an acetonitrile, a ketone, and an acetic-acid alkyl are mentioned. Although methyl acetate, ethyl acetate, an isopropyl acetate, n propyl acetate, an isobutyl acetate, etc. are illustrated and a methyl ethyl ketone, a methyl isobutyl ketone, etc. are illustrated as a ketone as an acetic-acid alkyl, a methyl ethyl ketone and a methyl isobutyl ketone are used preferably. Moreover, it is also possible to use other organic solvents together to these organic solvents. In this way, the polymerization of the polysiloxane which has a silanol group at the obtained chain end can be further carried out to the bottom of acid condensation catalyst existence. As an acid condensation catalyst, a concentrated sulfuric acid, a trifluoromethane sulfonic acid, dodecylbenzenesulfonic acid, etc. are illustrated. Moreover, what mixed 2-ethyl hexanoic acid with tetramethyl guanidine by this mol is effective. Since a condensation reaction is started at a room temperature and it is hard to start a siloxane chain scission, a trifluoromethane sulfonic acid is used preferably. Although especially the temperature requirement of a condensation reaction is not limited, it is preferably carried out by the 150-degree C temperature requirement from a room temperature. Although the example of a compound of this polysiloxane obtained by such method is shown, this invention is not limited to the following compounds. In addition, in compounds 1-6, n expresses one or more integers.

(Example 1 of a compound)

[0030]

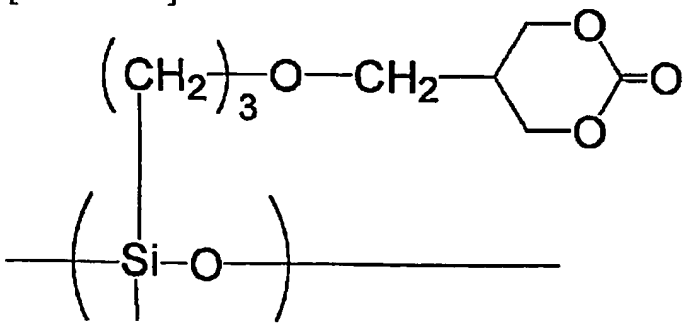
[Formula 20]

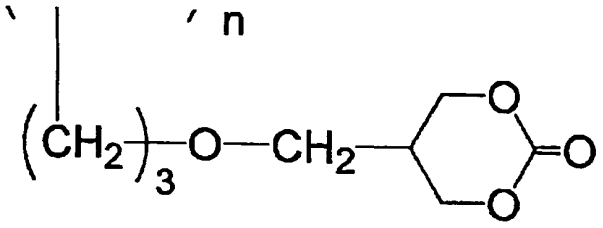


[0031] (Example 2 of a compound)

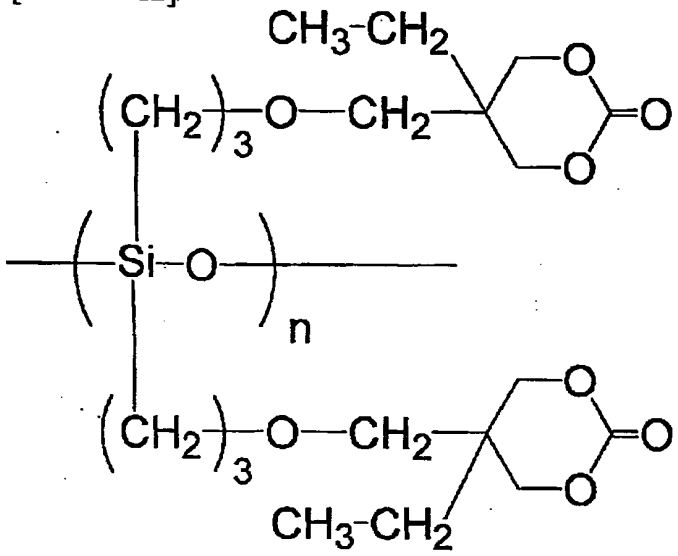
[0032]

[Formula 21]

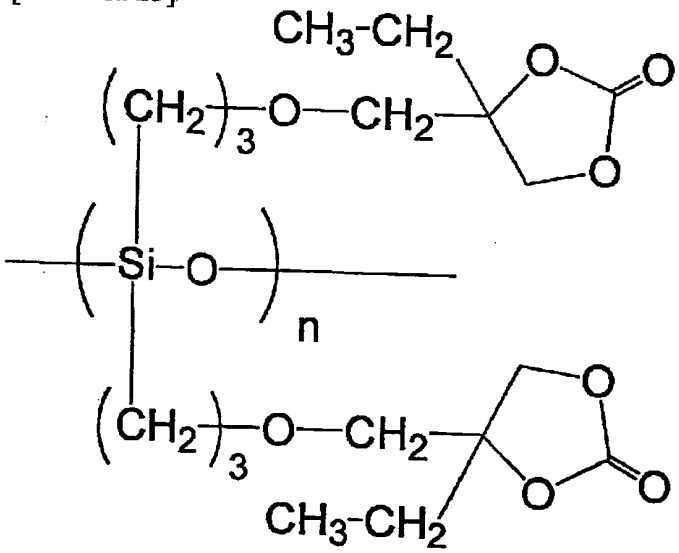




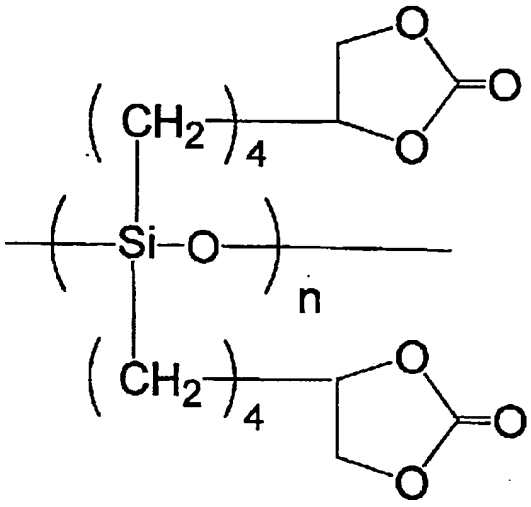
[0033] (Example 3 of a compound)
[0034]
[Formula 22]



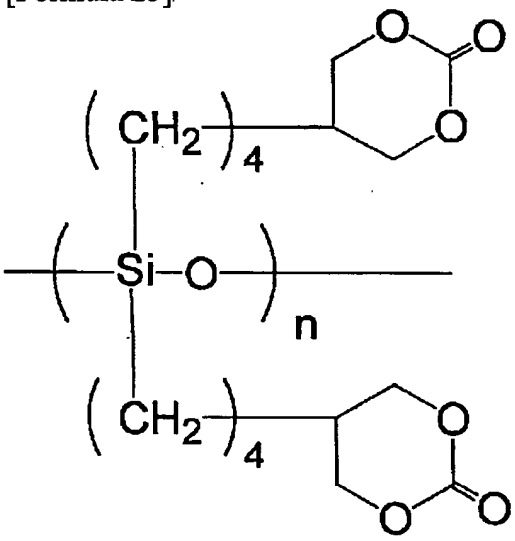
[0035] (Example 4 of a compound)
[0036]
[Formula 23]



[0037] (Example 5 of a compound)
[0038]
[Formula 24]



[0039] (Example 6 of a compound)
[0040]
[Formula 25]



[0041]
[Example] Next, although the example of this invention is explained concretely, this invention is not limited to the following examples.
(Example 1) To the autoclave made from stainless steel equipped with the glass container liner, 14.2g of annular (3-butenyl) ethylene ester carboxylic acids and 1.0mg (1.0x10-4mmol) of platinum vinyl siloxane xylene solutions were ****(ed), and in air, dichlorosilane 2.15g was added, and it heated and stirred at 60 degrees C at the autoclave. The reaction 6 hours after was ended and reaction mixture was distilled using the KUGERU roll. 5.65g (14.7mmol, 88% of yield) of dichlorosilane derivatives which, as a result, have two annular carbonate machines on silicon was obtained. Moreover, in the determination of molecular weight by GC-MS of this matter, the parent peak appeared in 385. Next, it was dropped at 1.22g of zinc oxides, and the reaction container of 5.0g of ethyl acetate into which it went, having dichlorosilane covered [which was obtained / 3.84g] it over 60 degrees C under heating stirring for 1 hour. Heating stirring was performed after that for further 3 hours. 20g of pure water after cooling was added, and it added until the solid content of a flask was lost in hydrochloric-acid water 10 more%. After it took out the organic layer and pure water washed enough, polysiloxane 3.15g was obtained by carrying out reduced pressure removal of the organic solvent. After having dissolved these polysiloxane 0.8g and 0.2g of lithium perchlorates in the

acetone, considering as the uniform solution and casting it on a substrate, it heated under argon gas atmosphere, the solvent was removed, and the thin film was obtained. The ionic conductivity of this thin film showed the very high value.

[0042]

[Effect of the Invention] The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is a new compound, and it has the feature that the manufacture method is simple and it is efficient.

[Translation done.]

* NOTICES *

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2. **** shows the word which can not be translated.
3.In the drawings, any words are not translated.

TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the polysiloxane which has two substituents which contain annular carbonate on a cell etc. on each silicon which may become an usable solid polymer electrolyte etc., and its manufacture method.

[Translation done.]

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PRIOR ART

[Description of the Prior Art] While resin industry develops in recent years, the resin which has a special property is needed and development of the macromolecule which is not in the former in connection with it has come to be desired. The uses of the polyether obtained by the ring opening polymerization of an epoxy compound, such as special rubber and a surfactant, are large. Moreover, the macromolecule which has a polyethylene oxide in a side chain can solubilize alkali-metal salts, such as lithium salt, and the research to a polyelectrolyte is prosperous in it. However, a polyethylene oxide cannot fully raise mobility of a metal salt for [the] high crystallinity.

[Translation done.]

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EFFECT OF THE INVENTION

[Effect of the Invention] The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is a new compound, and it has the feature that the manufacture method is simple and it is efficient.

[Translation done.]

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] this invention is to offer the new polysiloxane which has two substituents which contain annular carbonate on each silicon, and its manufacture method.

[Translation done.]

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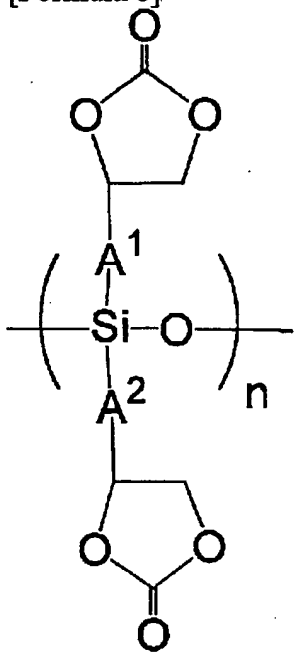
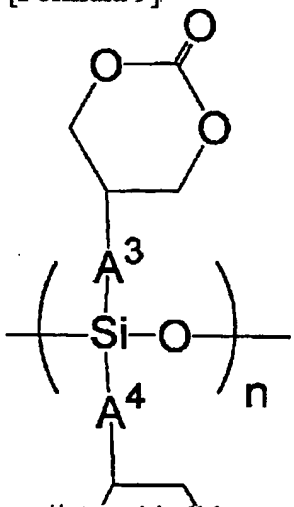
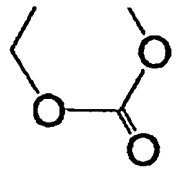
MEANS

[Means for Solving the Problem] this invention offers the new polysiloxane which has two substituents which contain annular carbonate on each silicon, and its manufacture method. Preferably, a principal chain is a general formula. [0005]
[Formula 8]

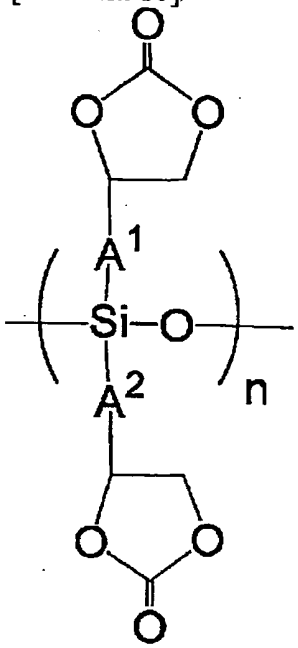


[0006] The polysiloxane of the above-mentioned publication which comes out and has two substituents which are shown, and which contain cyclic ether on each silicon (as for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine). Moreover, a principal chain is a general formula preferably.
[0007]
[Formula 9]

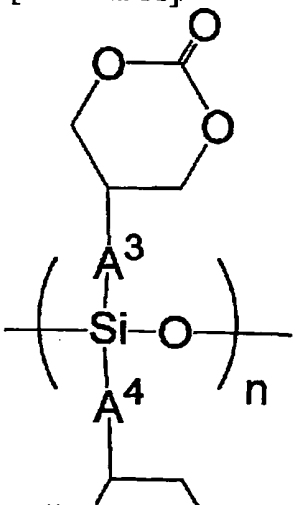


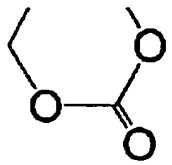


[0008] The polysiloxane of the above-mentioned publication which comes out and has two substituents which are shown, and which contain annular carbonate on each silicon (as for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine). Moreover, it is related with a polysiloxane including the siloxane unit which has two substituents which contain annular carbonate on each silicon, and its manufacture method. The polysiloxane which has the substituent which contains two annular carbonate on each of this silicon may contain other little siloxane units (the inside of a formula and R are substitution or an unsubstituted alkyl group), for example, R3SiO3/2 unit, and SiO4/2 unit. As a desirable thing, a principal chain is a general formula. [0009]
[Formula 10]

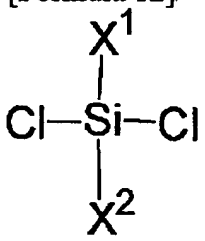


[0010] It can come out and the polysiloxane (as for the inside A1 and A2 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine) of the above-mentioned publication containing the siloxane unit which has two substituents which contain annular carbonate on each silicon shown can be mentioned. Moreover, as a desirable thing, a principal chain is a general formula. [0011]
[Formula 11]

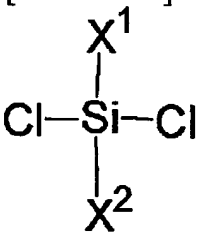




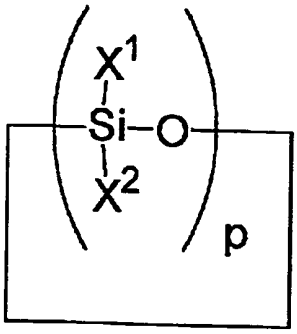
[0012] It can come out and the polysiloxane (as for the inside A3 and A4 of a formula, n expresses one or more integers for substitution or an unsubstituted alkyl group, an aryl group, or an oxy-alkylene machine) of the above-mentioned publication containing the siloxane unit which has two substituents which contain annular carbonate on each silicon shown can be mentioned. The manufacturing method of the above-mentioned polysiloxane is a method shown below. General formula. [0013]
[Formula 12]



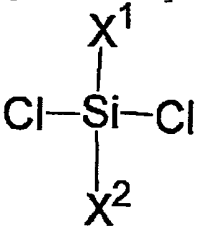
[0014] By stirring the dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) under existence of water by making a proton acid into a catalyst Chain both ends are made to generate the diorganopolysiloxane which has a silanol group. It is related with the new polysiloxane which is characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of acid condensation catalyst existence and which has two substituents which contain annular carbonate on each aforementioned silicon, and its manufacture method. Moreover, this invention is a general formula. [0015]
[Formula 13]



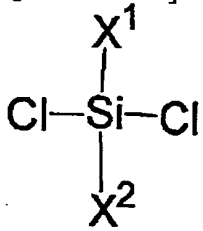
[0016] The dichlorosilane derivative shown by (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) Into the reaction mixture which the zinc oxide was made to react under existence of an organic solvent, and was subsequently obtained, for example, by stirring under existence of water by making into a catalyst the proton acid represented with a hydrochloric acid etc. Chain both ends are made to generate the diorganopolysiloxane which has a silanol group. It is related with the new polysiloxane which is characterized by making after an appropriate time carry out the condensation polymerization of this diorganopolysiloxane to the bottom of acid condensation catalyst existence and which has two substituents which contain an annular carbonate machine on each aforementioned silicon, and its manufacture method. Furthermore, this invention is a general formula. [0017]
[Formula 14]



[0018] (-- the inside X1 and X2 of a formula is the substituent which has an annular carbonate machine, X1 and X2 may be a homotypic or different species, respectively, and p is related with the manufacture method of a new polysiloxane of having two substituents which contain annular carbonate on each aforementioned silicon characterized by carrying out the polymerization of the cyclosiloxane derivative shown by 3 or 4) to the bottom of existence of a proton-acid catalyst
[0019]
[Embodiments of the Invention] If it hits enforcing the manufacture method of this invention, a zinc oxide or a proton acid is contacted in a suitable reaction container to this dichlorosilane. In this invention, the kind of reaction container is not important. However, in order to prevent side reaction, it is desirable to carry out in the reaction container formed with non-reactivity material. this invention method can be enforced with a batch method, a semi batch method, or continuous system. This reaction container is for example, a continuous stirring tank reaction container, and it deals in it. As for this method, it is desirable that a batch type or continuous system performs.
General formula. [0020]
[Formula 15]

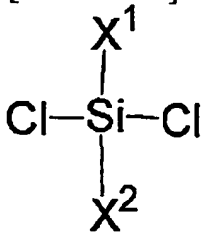


[0021] Although a zinc oxide contacts contact or a proton acid under existence of an organic solvent to the dichlorosilane shown by (the inside X1 and X2 of a formula may be the substituent which has an annular carbonate machine, and X1 and X2 may be a homotypic or different species, respectively), there are especially no restrictions in the contact method. The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is a general formula.
[0022]
[Formula 16]

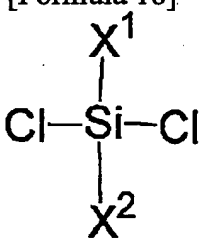


[0023] The dichlorosilane and the zinc oxide which are expressed with (the inside X1 and X2 of a formula may be the substituent which has an annular carbonate machine, and X1 and X2 may be a homotypic or different species, respectively) are made to react under existence of an organic solvent. Subsequently, although chain both ends are made to generate the diorganopolysiloxane which has a silanol group and the condensation polymerization of this diorganopolysiloxane is carried out subsequently to the bottom of existence of an acid condensation catalyst by adding and stirring water and a proton acid into the obtained reaction mixture The general formula which is a first train raw

material. [0024]
[Formula 17]



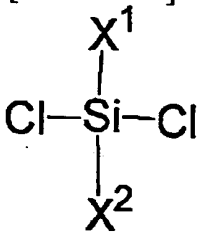
[0025] The dichlorosilane shown by (the inside X1 and X2 of a formula may be the substituent which has an annular carbonate machine, and X1 and X2 may be a homotypic or different species, respectively) is manufactured, for example by the following methods. It is obtained by contacting the end vinyl compound containing dichlorosilane (H2SiCl2) and an annular carbonate machine for the platinum compound catalyst or rhodium compound catalyst which is a catalyst for a hydrosilylation reaction, and carrying out an addition reaction. As an option which compounds this kind of dichlorosilane, there is the method of making the GURUNI yard reaction agent which corresponds to a tetrapod chlorosilicane (SiCl4) react, and acquiring, for example. The polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is the general formula manufactured by method which was described above. [0026]
[Formula 18]



[0027] The dichlorosilane and the zinc oxide which are expressed with (the inside X1 and X2 of a formula may be the substituent which has an annular carbonate machine, and X1 and X2 may be a homotypic or different species, respectively) are made to react under existence of an organic solvent. subsequently Although a molecule end is made to generate the diorganopolysiloxane which has a silanol group by added and stirring water and a proton acid into the obtained reaction mixture A zinc oxide and dichlorosilane react in an organic solvent, and produce a zinc chloride as a corresponding polysiloxane and a corresponding by-product here. Although especially limitation is not carried out, reaction temperature is preferably carried out in -30 degrees C - 200 degrees C, and is carried out under 50 degrees C - 150 degrees C overheating still more preferably. The amount of the zinc oxide used usually has desirable 0.5 to 4.0 which is the amount which becomes 0.4-5.0 mols per one mol of above-mentioned dichlorosilane, and is the equivalent mols here. As for the organic solvent used for this invention, an acetonitrile, a ketone, and an acetic-acid alkyl are mentioned. Although methyl acetate, ethyl acetate, an isopropyl acetate, n propyl acetate, an isobutyl acetate, etc. are illustrated and a methyl ethyl ketone and a methyl isobutyl ketone are illustrated as a ketone as an acetic-acid alkyl, an acetonitrile, methyl acetate, ethyl acetate, an isobutyl acetate, and a methyl isobutyl ketone are used preferably. Moreover, it is also possible to use other organic solvents together to these organic solvents. Then, by adding water and a proton acid, the polysiloxane which has a silanol group at the chain end is generated, this thing is dissolved in an organic-solvent layer, and the zinc chloride as a by-product dissolves in a water layer, and a superfluous zinc oxide also dissolves in a water layer, and it separates into two-layer. After an organic layer repeats washing with pure water, it is collected and dehydrated. The used solvent is removed by methods, such as reduced pressure removal. Furthermore, molecular weight can be increased using an acid condensation catalyst. As an acid condensation catalyst, a concentrated sulfuric acid, a trifluoromethane sulfonic acid, dodecylbenzenesulfonic acid, etc. are illustrated. Moreover, what mixed 2-ethyl hexanoic acid with tetramethyl guanidine by this mol is effective. Since a condensation reaction is started at a room temperature and it is hard to start a siloxane chain scission, a trifluoromethane sulfonic acid is used

preferably. Although especially the temperature requirement of a condensation reaction is not limited, it is preferably carried out by the 10 to 150 degrees C temperature requirement. Moreover, the polysiloxane which has two substituents which contain annular carbonate on each silicon of this invention is the general formula manufactured by method which was described above. [0028]

[Formula 19]

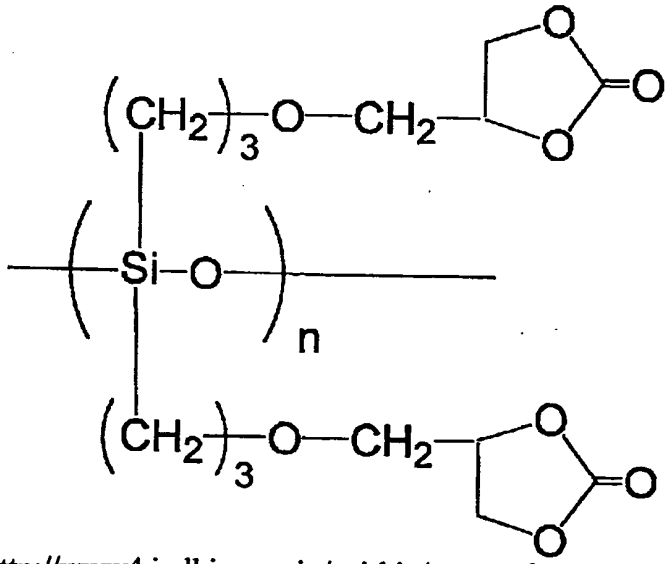


[0029] The dichlorosilane and the acid which are expressed with (the inside X1 and X2 of a formula being the substituent which has an annular carbonate machine, and being that X1 and X2 are of the same kind respectively or different species) are made to react under existence of an organic solvent. subsequently Although especially the acid used here although a molecule end is made to generate the diorganopolysiloxane which has a silanol group by added and stirring water and a proton acid into the obtained reaction mixture is not limited, a hydrochloric acid etc. is mentioned preferably. The amount of the acid used usually has desirable 0.5 to 4.0 which is the amount which becomes 0.4-5.0 mols per one mol of above-mentioned dichlorosilane, and is the equivalent mols here. As for the organic solvent used for this invention, diethylether, an acetonitrile, a ketone, and an acetic-acid alkyl are mentioned. Although methyl acetate, ethyl acetate, an isopropyl acetate, n propyl acetate, an isobutyl acetate, etc. are illustrated and a methyl ethyl ketone, a methyl isobutyl ketone, etc. are illustrated as a ketone as an acetic-acid alkyl, a methyl ethyl ketone and a methyl isobutyl ketone are used preferably. Moreover, it is also possible to use other organic solvents together to these organic solvents. In this way, the polymerization of the polysiloxane which has a silanol group at the obtained chain end can be further carried out to the bottom of acid condensation catalyst existence. As an acid condensation catalyst, a concentrated sulfuric acid, a trifluoromethane sulfonic acid, dodecylbenzenesulfonic acid, etc. are illustrated. Moreover, what mixed 2-ethyl hexanoic acid with tetramethyl guanidine by this mol is effective. Since a condensation reaction is started at a room temperature and it is hard to start a siloxane chain scission, a trifluoromethane sulfonic acid is used preferably. Although especially the temperature requirement of a condensation reaction is not limited, it is preferably carried out by the 150-degree C temperature requirement from a room temperature. Although the example of a compound of this polysiloxane obtained by such method is shown, this invention is not limited to the following compounds. In addition, in compounds 1-6, n expresses one or more integers.

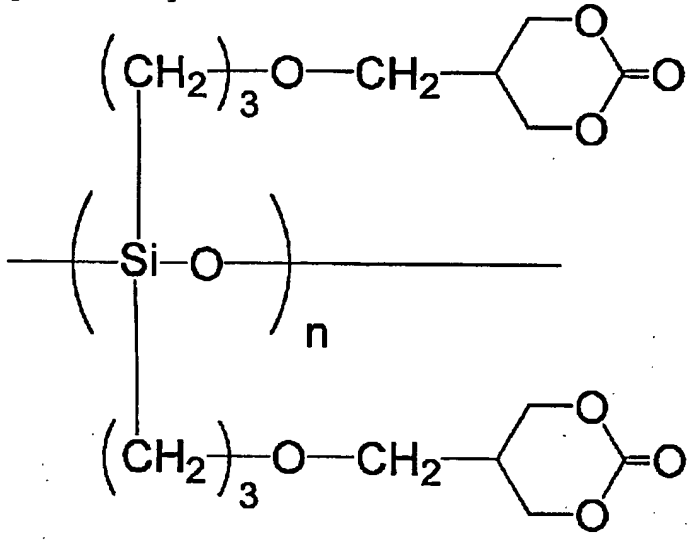
(Example 1 of a compound)

[0030]

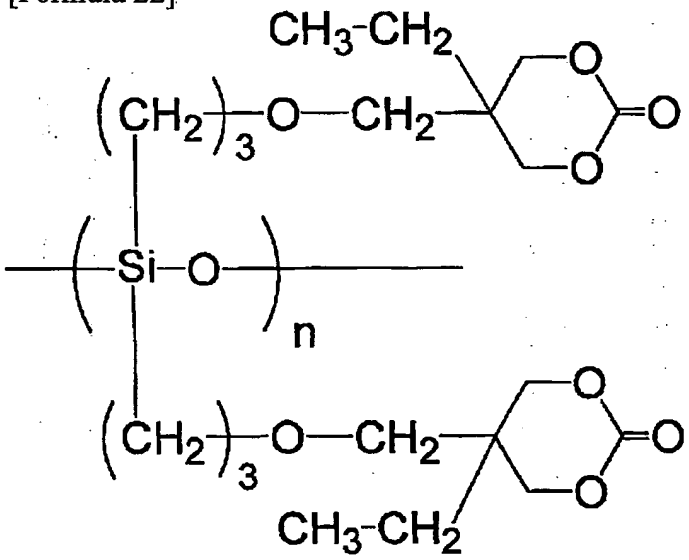
[Formula 20]



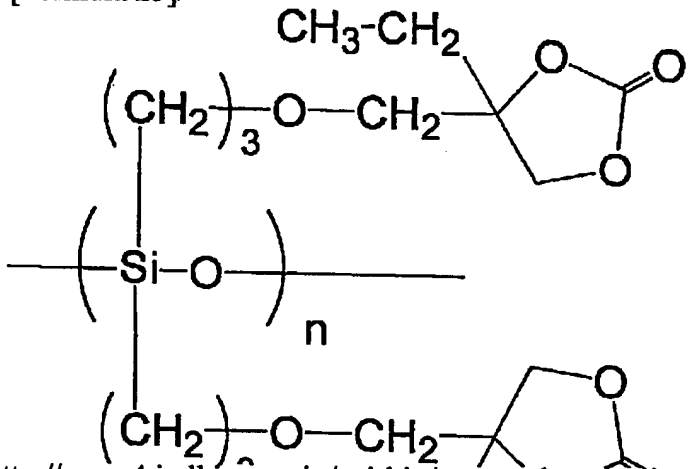
[0031] (Example 2 of a compound)
[0032]
[Formula 21]

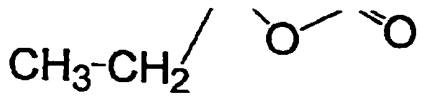


[0033] (Example 3 of a compound)
[0034]
[Formula 22]

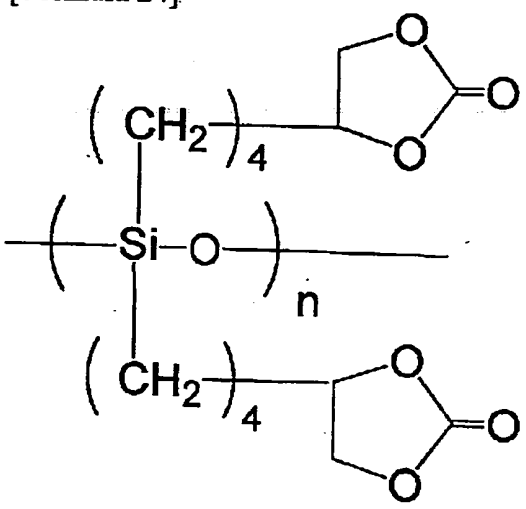


[0035] (Example 4 of a compound)
[0036]
[Formula 23]

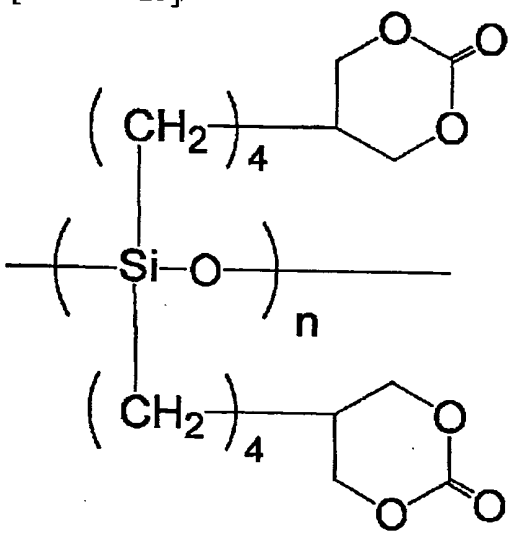




[0037] (Example 5 of a compound)
[0038]
[Formula 24]



[0039] (Example 6 of a compound)
[0040]
[Formula 25]



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EXAMPLE

[Example] Next, although the example of this invention is explained concretely, this invention is not limited to the following examples.
(Example 1) To the autoclave made from stainless steel equipped with the glass container liner, 14.2g of annular (3-butenyl) ethylene ester carboxylic acids and 1.0mg (1.0x10-4mmol) of platinum vinyl siloxane xylene solutions were ****(ed), and in air, dichlorosilane 2.15g was added, and it heated and stirred at 60 degrees C at the autoclave. The reaction 6 hours after was ended and reaction mixture was distilled using the KUGERU roll. 5.65g (14.7mmol, 88% of yield) of dichlorosilane derivatives which, as a result, have two annular carbonate machines on silicon was obtained. Moreover, in the determination of molecular weight by GC-MS of this matter, the parent peak appeared in 385. Next, it was dropped at 1.22g of zinc oxides, and the reaction container of 5.0g of ethyl acetate into which it went, having dichlorosilane covered [which was obtained / 3.84g] it over 60 degrees C under heating stirring for 1 hour. Heating stirring was performed after that for further 3 hours. 20g of pure water after cooling was added, and it added until the solid content of a flask was lost in hydrochloric-acid water 10 more%. After it took out the organic layer and pure water washed enough, polysiloxane 3.15g was obtained by carrying out reduced pressure removal of the organic solvent. After having dissolved these polysiloxane 0.8g and 0.2g of lithium perchlorates in the acetone, considering as the uniform solution and casting it on a substrate, it heated under argon gas atmosphere, the solvent was removed, and the thin film was obtained. The ionic conductivity of this thin film showed the very high value.

[Translation done.]